PROCEDURES FOR THE ANALYSIS OF EXPLOSIVES EVIDENCE

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These procedures have been reviewed and approved for use by the personnel of the Trace Evidence Section of the State Bureau of Investigation Crime Laboratory. This action does not signify this procedure to be mandated to the extent that it precludes the use of variations of this procedure or different procedures for accomplishing the desired assay. Physical and personnel resources, technological change, and examiner preference (within the bounds of good laboratory technique and quality control) determine what examination procedures are appropriate and / or acceptable for a given set of circumstances as encountered in the Trace Evidence Section.

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I. Name of Analytical Procedure

Explosives analysis.

II. Suggested Applications

Procedure for the examination of explosives evidence.

III. Introduction

Explosives evidence examined in the Trace Evidence Section is usually in one of two forms, pre-blast or post-blast. Pre-blast analysis involves the identification of an unexploded material that could range from anything such as gunpowder to C4. Analysis of these materials can be done in bulk form. However, in most cases, the analysis usually deals with the examination of post-blast materials or debris which requires the examination to be done on trace quantities of explosive and/or residual byproducts. This type of analysis employs the use of various techniques and instrumentation such as: visual examinations (macro and microscopic), odor assessments, ignition characteristics, spot tests, thin layer chromatography, infrared spectroscopy, polarizing light microscopy, scanning electron microscopy/x-ray analysis, other chromatographic methods (GC/MS), and x-ray diffraction.

IV. Analysis of pre-blast (intact) and post-blast explosives evidence

A. All outside packaging and its condition is noted.

B. The evidence container is opened and any distinctive odors are noted (odor assessment).

C. A macroscopic (visual) examination is performed as follows:

1. General appearance of the device/ debris is noted.

2. Locate and identify fragments/ components of the device (i.e. pipe/ container, blasting cap, electric matches, leg wires, wrappers, fuses, timing devices, batteries, etc.
3. Remove and isolate intact explosives.
    a. Note the location the explosives were found.
    b. Get an approximate weight of the intact explosive removed from the device, if feasible or requested.

D. A microscopic (stereomicroscope) examination is performed as follows:

1. Intact/ Unconsumed Particles – can use sieves to help isolate unconsumed particles from a post-blast device.
    a. Morphology (i.e. planed lumps, rough spheres, ball, flattened ball, disk, tube, perforated or not perforated, etc.
    b. Homogeneity (i.e. crystalline inclusions, metal flakes, sawdust, etc.
    c. Approximate size of particle(s)
    d. Presence of identification markers (i.e. Color - red dot, blue dot, green dot [smokeless powders])
    e. Ignition Characteristics

2. Consumed Particles/ Residues: Characterize and note the location of the residues (ex: white crystalline material present).

E. Separation of intact powders / residues

1. Manual (preferred): use tweezers or scalpel for manual separation.
2. Solvent Extraction – typical for residues
    a. Non-polar organic solvent (i.e. ether or pentane).
    b. Polar Organic Solvent (i.e. acetone or methanol)
    c. De-Ionized Water

F. Analytical Techniques

1. PLM (Polarizing Light Microscopy)
2. Spot tests (i.e. anions, cations)
    a. Diphenylamine
    b. Barium Chloride
    c. Silver Nitrate
    d. Anthrone
    e. Nessler's reagent
    f. Other
3. Cropen Test
4. TLC (Thin Layer Chromatography)
    a. Nitroglycerine
    b. Sugar & Chlorate
    c. Perchlorate / Chlorate / Nitrate

d. Other
5. FTIR (Fourier Transform Infrared Spectroscopy)
6. SEM/EDX (Scanning Electron Microscope/ Energy Dispersive X-Ray Spectroscopy)
7. XRD (X-Ray Diffraction)
8. GC (Gas Chromatography)
9. GC/MS (Gas Chromatography/ Mass Spectroscopy)
10. CE (Capillary Electrophoresis)

V. Chemical characterization of low explosives

A. Black Powder

1. Components to be identified
   a. Intact/ Unconsumed Particles
      i) Potassium Nitrate
      ii) Charcoal
      iii) Sulfur
   b. Residue / Common Solid Combustion Products
      i) K_2SO_4 - K_2CO_3
      ii) $KHSO_4$ - K_2S
      iii) KSCN – KHSO
      iv) S - KNO_3 - KNO_2
      v) $KHCO_3$ - $K_2S_2O_3$
      vi) C & Assoc. Ions
2. Methods suitable for characterization
   a. PLM
   b. Spot Tests
   c. FTIR
   d. TLC
   e. XRD
   f. SEM/EDX
   g. CE

B. Black Powder Substitute (Pyrodex)

1. Components to be identified
   a. Intact/ Unconsumed Particles
      i) Potassium Nitrate
      ii) Potassium Perchlorate
      iii) Charcoal
      iv) Sulfur
      v) Cyanoguanidine
      vi) Sodium Benzoate
      vii) Dextrine
      viii) Wax + Graphite

b. Residue / Common Solid Combustion Products
    i) K_2SO_4 - K_2CO_3
    ii) K_2S - S – KCl
    iii) $KHSO_3$ – KHSO
    iv) KNO_3 - $KClO_4$
    v) $KHCO_3$ - KNO_2
    vi) $K_2S_2O_3$ - C & Assoc. Ions
    vii) Cyanoguanidine
    viii) Sodium Benzoate

2. Methods suitable for characterization
    a. PLM
    b. Spot Tests
    c. FTIR
    d. TLC
    e. XRD
    f. SEM/EDX
    g. CE

C. Flash Powder (Pyrotechnic Powder)

1. General Flash Powder: Components to be identified
    a. Intact/ Unconsumed Particles
        i) Potassium Chlorate and/or Potassium Perchlorate
        ii) Aluminum
        iii) Sulfur
    b. Residue / Common Solid Combustion Products
        i) K_2SO_4 - $KClO_4$ or $KClO_3$ - $KHSO_3$ – KCl
        ii) $KHSO_3$ - Al_2O_3 - $AlCl_3$ - Al_2SO_4
        iii) $KAl(SO_4)_2$ - Al & Assoc. Ion
2. Military Flash Powder: Components to be identified
    a. Intact/ Unconsumed Particles
        i) Aluminum
        ii) Potassium Perchlorate
        iii) $Ba(NO_3)_2$
    b. Residue / Common Solid Combustion Products
        i) $KClO_4$ - KCl - Ba_2SO_4 - Al - Al_2O_3
        ii) $Ba(NO_3)_2$ - KNO_3 - $BaNO_2$ - $AlCl_3$ & Assoc. Ions
3. M-115: Components to be identified
    a. Intact/ Unconsumed Particles
        i) Magnesium
        ii) Aluminum
        iii) Potassium Perchlorate
    b. Residue / Common Solid Combustion Products
        i) KCl - Al_2O_3 - $KClO_4$ - Mg_2O_3 - & Assoc. Ions
4. M-117: Components to be identified
    a. Intact/ Unconsumed Particles

i) Magnesium
ii) Antimony Sulfide
iii) Potassium Perchlorate

b. Residue / Common Solid Combustion Products
i) KCl - Mg_2O_3 - Sb_2O_3 - $KClO_4$ & Assoc. Ions

5. M-119: Components to be identified
a. Intact/ Unconsumed Particles
i) Potassium Perchlorate
ii) Gallic Acid
iii) Red Gum

b. Residue / Common Solid Combustion Products
i) KCl

6. Methods suitable for characterization – all flash powders
a. PLM
b. Spot Tests
c. FTIR
d. TLC
e. XRD
f. SEM/EDX
g. CE

D. Smokeless Powders

1. Single-Base: components to be identified
a. Intact/ Unconsumed Particles
i) Nitrocellulose

b. Residue/ Common Solid Combustion Products
i) Nitrocellulose, (K_2SO_4 - KNO_2 weak)

2. Double-Base: components to be identified
a. Intact/ Unconsumed Particles
i) Nitrocellulose
ii) Nitroglycerin

b. Residue/ Common Solid Combustion Products
i) Nitrocellulose, (K_2SO_4 - KNO_2 weak)
ii) Nitroglycerin

3. Methods suitable for characterization – single and double base
a. PLM
b. Spot Tests
c. FTIR
d. TLC
e. GC or GC/MS

E. Sugar / Chlorate Mixtures
1. Components to be identified.
a. Intact/ Unconsumed Particles
i) A chlorate, such as Sodium Chlorate

ii) A sugar, such a sucrose

b. Residue / Common Solid Combustion Products

i) NaCl - $NaClO_3$ - Sucrose & Assoc. Ions

2. Methods suitable for characterization
   a. PLM
   b. Spot Tests
   c. FTIR
   d. TLC
   e. XRD
   f. SEM/EDX
   g. CE

F. Chemical Overpressure Devices (Acid / Foil Bomb)

1. Components to be identified.
   a. Intact/ Unconsumed Particles
      i) An acid, such as Hydrochloric Acid
      ii) Aluminum (foil)
   b. Residue / Common Solid Combustion Products
      i) Residual acid
      ii) Aluminum
      iii) Chloride ion
2. Methods suitable for characterization
   a. pH
   b. Spot Tests
   c. SEM/EDX

VI. Chemical characterization of high explosives

A. Organic high explosives

1. Organic high explosives are primarily nitroaromatics (TNT), nitramines (RDX), or nitrate esters (NG, PETN). They can be found as free flowing crystalline powders (PETN in detonating cord), solid case material (TNT booster), homogeneous mixtures (RDX and TNT in “military dynamite”) or in an organic matrix (RDX inC-4).
2. The following tests are used to identify the explosive in question:
   a. Visual / Microscopic Appearance - Describe color, consistency, and general physical appearance.
   b. Chemical Analysis - The sample is analyzed by FTIR, XRD, GC/MS, and/or TLC .
   c. Matrix or Binder - When appropriate, the polymeric matrix and/or plasticizer (binder) should be identified by using FTIR or GC/MS

B. Dynamite

1. Dynamites are considered only those formulations containing any combination of the organic nitrate esters NG, EGDN, MTN and DEGDN. In addition to the ingredients listed, common commercial dynamites contain carbonaceous filler material, inorganic oxidizing agents such as ammonium and sodium salts, and in

some instances sulfur, nitrocellulose, salts or microballoons.

2. The following tests are used to identify the explosive in question:
    a. Visual / Microscopic Appearance - Describe color, consistency, presence of prills, sulfur particles and fillers.
    b. Chemical analysis – the sample is analyzed by GC/MS or TLC

C. Blasting agents, slurries, and emulsions
1. Blasting Agents, Slurries and Emulsions include ANFO, binary explosives, water-gel, emulsions and a number of other explosives. The majority of these contain ammonium nitrate as the oxidizing agent combined with a sensitizer and/or fuel.
2. The following tests are used to identify the explosive in question:
    a. Visual / Microscopic Appearance - Describe color, consistency and general physical appearance.
    b. Chemical Analysis
        i) Inorganic oxidizing agent: confirmed by the use of SEM/EDX, CE, XRD and spot tests with appropriate confirmatory analysis.
        ii) Sensitizer:
            a) If present, is typically an amine salt (such as ethylene diamine dinitrate, monomethylamine nitrate or hexamine nitrate), aluminum powder or microballoons.
            b) Identified using TLC or GC/MS
        iii) Fuel:
            a) If present, typically a petroleum distillate such as diesel in ANFO, nitromethane in binary explosives or a high molecular weight oil or wax
            b) Identified by FTIR or GC/MS.

D. Primary (initiating) high explosives
1. The primary high explosives are those materials sensitive to shock, flame and friction typically composed of styphnates, azides, fulminates, organic diazo compounds (diazodinitrophenol), HMTD, TATP, or other organic peroxides.
2. The following tests are used to identify the explosive in question:
    a. Visual / Microscopic Appearance - Describe color and general physical appearance.
    b. Ignition Susceptibility Test (Flame Test) - When appropriate, a VERY small particle is ignited and the burning characteristics are described.
    c. Chemical Analysis - FTIR or XRD is sufficient to identify most primary explosives. In some situations a chromatographic or second appropriate instrumental analysis (TLC or XRF) may be required. Mass spectrometry is useful for peroxide explosives.

E. In cases involving unusual or novel explosives where reference materials are not available, additional examinations may be necessary to fully characterize the material.

VII. Criteria for the identification of explosives

A. Low Explosives: Intact / Unconsumed Particles

1. The material in question should be characterized physically and chemically.
2. Physical characteristics such as general appearance, color, morphology, presence of markers, homogeneity, the presence of catalysts such as metal, particle size, and ignition susceptibility should be documented.
3. Chemical characterization
    a. Should be performed and documented by at least two or more of the following methods:
        i) PLM
        ii) Spot Tests
        iii) pH
        iv) FTIR
        v) TLC
        vi) SEM/EDX
        vii) XRD
        viii) GC or GC/MS
    b. The number of methods for chemical characterization will vary depending on the type of propellant encountered.
4. If the physical and chemical characteristics do not correspond to the formulation of known propellants then no identification should be made.

B. Low Explosives: Post-combustion

1. The material in question should be characterized physically and chemically.
2. Chemical characterization
    a. Should be performed and documented by at least two or more of the following methods:
        i) PLM
        ii) Spot Tests
        iii) pH
        iv) FTIR
        v) TLC
        vi) SEM/EDX
        vii) XRD
        viii) GC or GC/MS
        ix) CE

    b. The number of methods for chemical characterization will vary depending on the type of propellant encountered.

C. Criteria for the identification of intact / bulk high explosives

1. The material in question should be characterized physically and chemically.
2. Physical characteristics such as general appearance, color, morphology, presence of markers, prills, or microballoons, consistency, homogeneity, presence of catalysts such as metal, particle size, and ignition susceptibility should be documented.
3. Chemical characterization

a. Should be performed and documented by at least two or more of the following methods:
   i) PLM
   ii) Spot Tests
   iii) FTIR
   iv) TLC
   v) SEM/EDX
   vi) XRD
   vii) GC or GC/MS
   viii) CE

b. The number of methods for chemical characterization will vary depending on the type of high explosive encountered.

4. If the physical and chemical characteristics do not correspond to the formulations of known high explosives, then no identification should be made.

VIII. Conclusions for explosives analysis

A. Explosive Materials prior to deflagration/detonation (Intact)

1. Positive Conclusion

a. In most cases an identification of the explosive material will be determined in conjunction with a known standard (if available).

b. Example: Examination of Item #1 revealed the presence of Composition C-4.

2. Negative Conclusion

a. Example: Examination of Item #1 failed to reveal the presence of explosives.

3. Statements regarding functionality may also be included.

B. Explosive Residues (Post-Blast)

1. Positive Conclusion

a. Conclusions may include explosive materials, common solid combustion products, and non-explosive components. Statements of functionality may be included.

b. Example: Examination of Item #1 revealed the presence of a damaged cardboard tube, burned safety fuse, and a gray powder residue. The gray

powder residue contained aluminum, sulfur, potassium sulfate, and aluminum sulfate, which is consistent with the presence of post-combustion flash powder. These materials are consistent with having come from a post-blast improvised explosive device.

2. Negative Conclusion

Example: Examination of Item #1 failed to reveal the presence of explosives.

C. Hoax Devices

Example: Examination of Item # 1 revealed the presence of a metal pipe nipple, two end caps, and a union with electrical wires attached. This item is not an explosive device, however it is consistent in appearance to an explosive device and would be deemed a hoax explosive device.

Revision History

Effective Date	Reason
03-10-2010	Updated conclusion examples, updated spot tests, removed TLC, separated Chemical Overpressure Devices into its own section, and changed document flow.

Appendix I:
Spot Test Reagent Preparation

All spot test reagents are prepared as listed in the technical procedure. Reagents do not expire unless otherwise noted. The reagent may continue to be used as long as it passes its QC check prior to each use and is not expired.

A. Diphenylamine – test for nitrates
   1. To prepare, dissolve 1 mg of diphenylamine in 10 mL of concentrated sulfuric acid.
   2. Expires 1 year from date of preparation
   3. The solution is QC checked with a known nitrate (i.e. KNO_3). If the solution turns blue in the presence of the known nitrate, this is considered a positive QC check.

B. Barium Chloride – test for sulfates
   1. To prepare, dissolve 5 g of barium chloride in 100 mL of water.
   2. Does not expire.
   3. The solution is QC checked with a known sulfate. If a white precipitate is formed, this is considered a positive QC check.

C. Silver Nitrate – test for chlorides
   1. To prepare, dissolve 5 g of silver nitrate in 100 mL of water.
   2. Does not expire
   3. The solution is QC checked with a known chloride. If a white precipitate is formed, this is considered a positive QC check.

D. Anthrone – test for sugars
   1. Does not expire.
   2. This test is prepared for each examination. It is QC checked by adding a known sugar to anthrone crystals in a spot plate. Three drops of sulfuric acid are added. If the solution turns a blue to blue-green color in the presence of the known sugar, this is considered a positive QC check.

E. Nessler's reagent – test for ammonium
   1. A commercially purchased Nessler's reagent will be used.
   2. This reagent expires according to the manufacture's recommendations.
   3. The solution is QC checked with a known ammonium containing compound (i.e. ammonium nitrate). An orange-brown color formation in the presence of a known ammonium cation, is considered a positive QC check.

Appendix II:
Modified test for chlorates and perchlorates (Cropen Test)

A. Reagent preparation
   1. Solution A: 5 grams of Zinc Sulfate and 4 grams of Potassium Nitrate in 40 milliliters of water
   2. Solution B: 0.015% Methylene Blue
   3. QC Check: Crystals as described below form in the presence of known chlorates and perchlorates.
   4. These solutions do not expire.

B. Test Procedure:
   1. Place a droplet of solution A and a droplet of solution B next to each other, but not touching on a microscope slide mounted on a polarizing light microscope (PLM).
   2. Add a small amount of the material to be tested to the solution A droplet.
   3. With a glass rod or probe, draw droplet B to droplet A until they touch.
   4. 4 - Examine the interface area for the formation of any crystals.

C. Crystal descriptions
   1. Perchlorate Crystals
      Blue needles, some with a purplish tinge growing singly and in bundles. The crystals are pleochroic. They are dark blue perpendicular and pale blue parallel to the needle length. As they grow they start to develop blunt and sometimes split ends. They are also anisotropic with parallel extinction.
   2. Chlorate Crystals
      Mainly blue rosettes (sometimes with a slight purple tinge) develop at a slower rate than the perchlorate, however some single crystals will form. The chlorate crystals are smaller and the needles are thinner than the perchlorate form. The needles are pleochroic and isotropic as those formed with perchlorates.

Appendix III:
Test for acids and bases (pH paper)

A. Materials Required
   1. pH paper
   2. pipette

B. Test Procedure
   1. Place a droplet of unknown solution on full range pH test paper.
   2. Observe color change while the pH paper is still wet and compare to scale on pH test paper container.
   3. Record reading from scale comparison.
   4. This procedure may be repeated using a narrow range pH paper to further classify the acidity or basicity of the liquid.

C. Conclusions
   1. If the pH is less than 7, the solution is acidic.
   2. If the pH is greater than 7, the solution is basic.
   3. A pH equal to 7 is neutral.
   4. Strong acids will have a pH of 0.
   5. Strong bases will have a pH of 13 or greater.

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General Flow Diagram for Post-Blast Explosives Identification